National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

PIC Phase II Proposal

Project 2

Title: Advanced Instrumented Indentation Polymeric Materials

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Objective: To advance the metrology of instrumented indentation on polymeric systems.

- Further identify and minimize uncertainties for shallow depth indents.
- Establish viscoelastic standards to evaluate and improve viscoelastic measurement methods.
- Explore use of confinement in thin multi-layered coatings for increased indention and scratch resistance.
- Include contributions of adhesive forces.
- Apply knowledge gained from real model systems to multi-component, nano-composite polymeric systems.
- Apply advanced metrology of instrumented indentation toward goal of quantitative scratch and mar method.

Motivations

- Emerging Field
  - Web of Science (ISI)
    - ~1700 papers refer to nanoindentation
    - ~ only 70 of those include nanoindentation of polymers
- NIST & Nanotechnology Initiative
    - Develop traceable measurements, standards, reference materials, data, etc., that will enable the infrastructure to facilitate commercialization
- Nanometrology
  - Fundamental need to understand nanoscale properties related to overall performance
- Significant progress
    - Technology improving monthly
    - Multidisciplinary
      - Contact mechanics
      - Tribology
      - Rheology
      - Science and Engineering
    - Opportunity
      - Improve the service life, performance, design of polymeric materials
      - Real industrial impact
      - competitive advantage

Approach

1. **Recognize and Minimize**
   1. Error contributions (frame compliance, load calibration, dynamic calibrations, low s/n ratios, tip defects, tip shapes, surface roughness, etc.).
2. **Magnitude**
   1. Use model, well characterized nearly monodisperse polymeric systems.
   2. Compare indentation results to well characterized bulk measurements and JKR-type Hertzian contact experiments.
3. **Sensitivity**
   1. Explore ability to measure differences in relaxation behavior and mechanical properties via changes in tacticity, MW, and temperature in model polymeric systems.
4. **Improvements**
   1. Recommend improvements in test method, instrument dynamic calibration and instrument design.
5. **Application**
   1. Apply improved metrology to industrial materials (i.e. polydisperse systems, additives, fillers, pigments, multi-component, etc.).

Milestones

1. Purchase additional spherical and conical tips. Conduct tip reconstruction for accurate A(hc) data. Complete indentation and rheological-type indentation measurements on model polymer systems. Report novel new measurements, report additional ways to improve measurements.

2. Compare magnitude and sensitivity of indentation results to bulk indentation and viscoelastic properties on model systems. Collaborate with academic colleagues in instrumented viscoelasticity to aid in improvement of viscoelastic characterization. Report conclusions.

3. Develop methodologies for measuring weak adhesion with the nanoindenter.

3. Create and measure indention and scratch resistance of model multi-layered samples. Work with academic collaborations to create novel interfacial patterned and confined films for increased scratch and indentation resistance.

4. Apply improved metrology to more complex model block copolymer and polymer blend systems. Characterize industrial relevant polymers.

Impact

- Significantly advance instrumented indentation for the analysis of nano-mechanical properties of polymeric systems with the goal of developing a science-based methods to provide mechanical properties of polymeric materials at the micro-nanoscale.